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# **PCT**

# REQUEST

	For receiving Office use only	
International Applic	ation No.	
International Filing	Date	
Name of receiving (	Office and "PCT International	Application"

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.	Name of receiving Offi	ce and "PCT International Application"
	Applicant's or agent's file reference (if desired) (12 characters maximum) PC3-160	
Box No. I TITLE OF INVENTION Production Processes and Systems, Compositions Surfactants. Monomer Units, Metal Complexes, Phosphate Esters Glycols Aqueous Film Forming Foams and Foam Stabilizers		
Box No. II APPLICANT This person	is also inventor	
Name and address: (Family name followed by given name; for a legal ent. The address must include postal code and name of country. The country of t. Box is the applicant's State (that is, country) of residence if no State of reside.	he address indicated in this	Telephone No. 302-552-3103
PCBU Services, Inc.		Facsimile No.
300 Delaware Ave., Suite 1269		302-552-3128
Wilmington, DE 19801		Teleprinter No.
		Applicant's registration No. with the Office
State (that is, country) of nationality:	State (that is, country)	of residence:
United States	Delaware	
		the United States indicated in the States indicated in the Supplemental Box
Box No. III FURTHER APPLICANT(S) AND/OR (FURT	HER) INVENTOR(S)	
Name and address: (Family name followed by given name; for a legal ent The address must include postal code and name of country. The country of Box is the applicant's State (that is, country) of residence if no State of reside. BOGGS, Janet 6925 N. 900 East Brownsburg, IN 46112	he address indicated in this	This person is:  applicant only  applicant and inventor  inventor only (If this check-box is marked, do not fill in below.)  Applicant's registration No. with the Office
State (that is, country) of nationality:  State (that is, country) of residence:		
United States	United States	
This person is applicant for the purposes of:  all designated the United States all designated the United States	d States except tates of America	the United States of America only the States indicated in the Supplemental Box
Further applicants and/or (further) inventors are indicated of	on a continuation sheet.	
Box No. IV AGENT OR COMMON REPRESENTATIVE		CORRESPONDENCE
The person identified below is hereby/has been appointed to act of the applicant(s) before the competent International Authorities	as:	agent common representative
Name and address: (Family name followed by given name: for a legal ent The address must include postal code and name of	country.)	Telephone No. 509-624-4276
HYTA, Robert C.; TAYLOR, Jennifer J.; MA		Facsimile No.
MALHOTRA, Deepak; HENDRICKSEN, Ma GRIGEL, George G.; LATWESEN, David G	•	509-838-3424
Keith D.; SHAURETTE, James D.; LAKE Ja		Teleprinter No.
KENADY D. Brent; all of Wells St. John P.S		Agent's registration No. with the Office
First Ave, Suite 1300, Spokane, WA 99201-	3828, USA	
Address for correspondence: Mark this check-box where space above is used instead to indicate a special address to		

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)		
If none of the following sub-boxes is used, this sheet should not be included in the request.		
Name and address: (Family name followed by given name; for a legal entithe address must include postal code and name of country. The country of the Box is the applicant's State (that is, country) of residence if no State of resider BRANDSTADTER, Stephan M. 3946 N. Washington Blvd. Indianapolis, IN 47906	ne address indicated in this	
State (that is, country) of nationality: United States	State (that is, country) of residence: United States	
This person is applicant all designated states all designated the United St	States except ates of America of America only the States indicated in the Supplemental Box	
Name and address: (Family name followed by given name: for a legal entite the address must include postal code and name of country. The country of the Box is the applicant's State (that is, country) of residence if no State of residence that is, and the applicant's State of the country of the applicant's State (that is, country) of residence if no State of residence that the applicant is state of the address of the applicant is state of the address of the applicant is state of the address of the	e address indicated in this	
State (that is, country) of nationality: United States	State (that is, country) of residence: United States	
This person is applicant all designated all designated for the purposes of:	I States except atte United States the States indicated in the Supplemental Box	
Name and address: (Family name followed by given name; for a legal entit The address must include postal code and name of country. The country of the Box is the applicant's State (that is, country) of residence if no State of residence SHARMA, Vimal 2101 Cumberland Ave, 5104 West Lafayette, IN 47906	e address indicated in this	
State (that is, country) of nationality: United States	State (that is, country) of residence: United States	
This person is applicant all designated all designated	States except ates of America only the States indicated in the Supplemental Box	
Name and address: (Family name followed by given name; for a legal entity The address must include postal code and name of country. The country of the Box is the applicant's State (that is, country) of residence if no State of residence EDWARDS, E. Bradley 3704 Woodcliff Drive Lafayette, IN 47905	e address indicated in this	
State (that is, country) of nationality: United States	State (that is, country) of residence: United States	
	States except attes of America only the States indicated in the Supplemental Box	
Further applicants and/or (further) inventors are indicated or	n another continuation sheet.	

Form PCT/RO/101 (continuation sheet) (April 2005)

See Notes to the request form

Supplemental Box If the Supplemental Box is not used, this sheet should not be included in the request.

- If, in any of the Boxes, except Boxes Nos. VIII(i) to (v) for which a special continuation box is provided, the space is insufficient to furnish all the information: in such case, write "Continuation" HEDRICK Victoria of Box No .... " (indicate the number of the Box) and furnish the information in the same manner as required according to the captions of the Box in which the space was insufficient, in
- if more than two persons are to be indicated as applicants and/or inventors and no "continuation sheet" is available: in such case, write "Continuation of Box No. III" and indicate for each additional person the same type of information as required in Box No. III. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below;
- if, in Box No. II or in any of the sub-boxes of Box No. III, the indication "the States indicated in the Supplemental Box" checked: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Boxes No. II and No. III" (as the case may be), indicate the name of the applicant(s) involved and, next to (each) such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is applicant;
- if, in Box No. II or in any of the sub-boxes of Box No. III, the inventor or the inventor/applicant is not inventor for the purposes of all designated States or for the purposes of the United States of America: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Box No. II and No. III" (as the case may (iii) be), indicate the name of the inventor(s) and, next to (each) such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is inventor;
- if, in addition to the agent(s) indicated in Box No. IV, there are (iv) further agents: in such case, write "Continuation of Box No. IV" and indicate for each further agent the same type of information as required in Box No. IV;
- if, in Box No. VI, there are more than three earlier applications whose priority is claimed: in such case, write "Continuation of Box No. VI" and indicate for each additional earlier application the same type of information as required in Box No. VI.
- If the applicant intends to make an indication of the wish that the international application be treated, in certain designated States, as an application for a patent of addition, certificate of addition, inventor's certificate of addition or utility certificate of addition: in such a case, write the name or two-letter code of addition: in such a case, write the name or two-letter code of each designated State concerned and the indication "patent of addition," "certificate of addition," "inventor's certificate of addition" or "utility certificate of addition," the number of the parent application or parent patent or other parent grant and the date of grant of the parent patent or other patent grant or the date of filing of the parent application (Rules 4.11(a)(iii) and (Obis 1/a) or (b)) and 49bis. I(a) or (b)).
- 3. If the applicant intends to make an indication of the wish that the international application be treated in the United States of America, as a continuation or continuation-in-part of an earlier application: in such a case, write "United States of America" or "US" and the indication "continuation" or "continuation-in-part" and the number and the filing date of the parent application (Rules 4.11(a)(iv) and 49bis.1(d)).

HEDRICK, Victoria 9262 South 300E Brookston, IN 47923 State of Nationality: US State of Residence: US

Applicant and Inventor: Applicant for US only

JACKSON, Andrew 3346 Whirl Away Court West Lafayette, IN 47906 State of Nationality: US State of Residence: US

Applicant and Inventor: Applicant for US only

LEMAN, Gregory 3319 Crawford Street West Lafayette, IN 47906 State of Nationality: US State of Residence: US

Applicant and Inventor: Applicant for US only

NORMAN, Edward 942 St. Matthews Road Chester Springs, PA 19425 State of Nationality: US State of Residence: US

Applicant and Inventor: Applicant for US only

KAUFMAN, Robert 8129 Stanford Ave St. Louis, MO 63130 State of Nationality: US State of Residence: US

Applicant and inventor: Applicant for US only

### Box No. VIII (ii) DECLARATION: ENTITLEMENT TO APPLY FOR AND BE GRANTED A PATENT

The declaration must conform to the standardized wording provided for in Section 212; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No.VIII (ii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51bis.1(a)(ii)), in a case where the declaration under Rule 4.17(iv) is not appropriate:

in relation to this international application no. PCT/US05/03138.

PCBU Services, Inc. is entitled to apply for and be granted a patent by virtue of the following:

an assignment from:

BOGGS, Janet, 6925 N. 900 East, Brownsburg, IN 46112, United States of America, dated 3 May 2005 (03.05.2005);

BRANDSTADTER, Stephan M., 3946 N. Washington Blvd, Indianapolis, IN 47906, United States of America, dated 9 May 2005 (09.05.2005);

CHIEN, John, 3318 Humboldt Street, West Lafayette, IN 47906, United States of America, dated 3 May 2005 (03.05.2005);

SHARMA, Vimal, 2101 Cumberland Ave 5104, West Lafayette, IN 47906, United States of America, dated 9 May 2005 (09.05.2005);

EDWARDS, E. Bradley, 3704 Woodcliff Drive, Lafayette, IN 47905, United States of America, dated 3 May 2005 (03.05.2005);

HEDRICK, Victoria, 9262 South 300E, Brookston, IN 47923, United States of America, dated 3 May 2005 (03.05.2005);

JACKSON, Andrew, 3217 Hopkins Court, West Lafayette, IN 47906, United States of America, dated 3 May 2005 (03.05.2005);

LEMAN, Gregory, 3319 Crawford Street, West Lafayette, IN 47906, United States of America, dated 3 May 2005 (03.05.2005);

NORMAN, Edward, 942 St. Matthews Road, Chester Springs, PA 19425, United States of America, dated 24 May 2005 (24.05.2005);

KAUFMAN, Robert, 8129 Stanford Ave, St. Louis, MO 63130, United States of America, dated 12 May 2005 (12.05.2005).

This declaration is made for the purposes of all designations.

This declaration is continued on the following sheet, "Continuation of Box No. VIII (ii)"
---

### Box No. VIII (iii) DECLARATION: ENTITLEMENT TO CLAIM PRIORITY

The declaration must conform to the standardized wording provided for in Section 213; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No.VIII (iii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application specified below, where the applicant is not the applicant who filed the earlier application or where the applicant's name has changed since the filing of the earlier application (Rules 4.17(iii) and 51bis.1(a)(iii)):

in relation to international application no. PCT/US05/03138.

PCBU Services, INC. is entitled to claim priority to earlier application serial no. 60/540,612, filed 30 January 2004 (30.01.2004) in the United States of America by virtue of the following:

an assignment from BRANDSTADTER, Stephan M. and SHARMA, Vimal, dated 9 May 2005 (09.05.2005);

an assignment from BOGGS, Janet, CHIEN, John, EDWARDS, E. Bradley, HEDRICK, Victoria, JACKSON, Andrew, and LEMAN, Gregory, dated 3 May 2005 (03.05.2005);

an assignment from KAUFMAN, Robert dated 12 May 2005 (12.05.2005); and

an assignment from NORMAN, Edward dated 24 May 2005 (24.05.2005).

This declaration is made for the purposes of all designations.

This declaration is continued on the following sheet, "Continuation of Box No. VIII (iii	iii)"
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Box No. VIII (iv) DECLARATION: INVENTORSHIP (only for the purposes of the designation of the United States of America)
The declaration must conform to the following standardized wording provided for in Section 214; see Notes to Boxes Nos. VIII, VIII (I) to (v)
(In general) and the specific Notes to Box No. VIII (iv). If this Box is not used, this sheet should not be included in the request.

Declaration of inventorship (Rules 4.17((v) and 51 bis.1(a)((v)) for the purposes of the designation of the United States of America:
I hereby declare that I believe I am the original, terst and sole (if only one inventor is listed below) or joint (if more than one inventor is listed below) inventor of the subject matter which is claimed and for which a patent is sought.
This declaration is directed to the international application of which it forms a part (if filing declaration with application).
This declaration is directed to international application No. PCT/
I hereby declare that my residence, mailing address, and citizenship are as stated next to my name.
I hereby state that I have reviewed and understand the contents of the above-identified international application, including the claims of said application. I have identified in the request of said application, in compliance with PCT Rule 4.10, any claim to foreign priority, and I have identified below, under the heading "Prior Applications," by application number, country or Member of the World Trade Organization, day, month and year of filing, any application for a patent or inventor's certificate filed in a country other than the United States of America, including any PCT international application designating at least one country other than the United States of America, having a filing date before that of the application on which foreign priority is claimed.
Prior Applications: US No. 60/540,612 filed 30 January 2004 (30.01.04) entitled "Fluorine Functional Groups, Fluorine Compositions, Processess for Manufacturing Fluorine Compositions, and"
I hereby acknowledge the duty to disclose information that is known by me to be material to patentability as defined by 37 C.F.R. § 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the PCT international filing date of the continuation-in-part application.
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful fulse statements may jeopardize the validity of the application or any patent issued thereon.
Name: BOGGS, Janet,
Residence: Brownsburg, IN, United States (city and either US state, if applicable, or country)
Mailing Address: -6022.N., 990 East, Brownsburg, IN. 46112, United States of America
Citizenship: United States
Inventor's Signature. The filing of the international application. The signature must be that of the inventor, not that of the agent)  Dute: (of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26ter after the filing of the international application)
BRANDSTADTER, Stephan M.
Residence: Indianapolis, IN, United States (city and either US state, if applicable, or country)
Mailing Address: 3946 N. Washington Blvd., Indianapolis, IN 47906
Citizenship: United States
Inventor's Signature:  (if not contained in the request, or if declaration is corrected or (of signature which is not contained in the request, or of the

This declaration is continued on the following sheet, "Continuation of Box No. VIII (iv)".

the agent)

added under Rule 26ter after the filing of the international

application. The signature must be that of the inventor, not that of

declaration that is corrected or added under Rule 26ter after the

filing of the international application)

Continuation of Box No. VIII (i) to (v) DECLARATION  If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two invents are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII" (indicate the item number of the Box) and furn the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is near the respect of two or more declarations, a separate continuation box must by used for each such declaration. If this Box is not used, this should not be included in the request.
Continuation of Box No. VIII(iv) Name: CHIEN, John Residence: West Lafayette, IN, United States Mailing Address: 3318 Humbolt Street, West Lafayette, IN 47906, US Citizenship: United States
Name: SHARMA, Virnal
Name: EDWARDS, E. Bradley
Name: HEDRICK, Victoria
Name: JACKSON, Andrew
Name: LEMAN, Gregory
Name: NORMAN, Edward Edward C. Vum 5/25/05 Residence: Chester Springs, PA, United States Mailing Address: 942 St. Matthews Road, Chester Springs, PA 19425, US Citizenship: United States
Name: KAUFMAN, Robert Robert Court Court May 24, 2007 Residence: St. Louis, MO, United States Mailing Address: 8129 Stanford Ave, St. Louis, MO 63130 Citizenship: United States

Continuation of Box No. VIII (I) to (v) DECLARATION  If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the cases are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII" (Indicate the Item the information in the same manner as required for the purposes of the Box in which the space was insufficient in respect of two or more declarations, a separate continuation box must be used for each such declaration. If should not be included in the request.	where more than two inventors number of the Box) and furnish t. If additional space is needed (this Box is not used, this sheet
Continuation of Box No. VIII(iv)  Name: CHIEN, John  Residence: West Lafayette, IN, United States  Mailing Address: 3318 Humbolt Street, West Lafayette, IN 47906, US  Citizenship: United States Humbold t	os:
Name: SHARMA, Vimal	D6, US,
Name: EDWARDS, E. Bradley S. Stading Sdwards Residence: Lafayette, IN, United States Mailing Address: 3704 Woodcliff Drive, Lafayette, IN 47905, US Citizenship: United States	52505
Name: HEDRICK, Victoria Victoria Gladuck Residence: Brookston, IN, United States Mailing Address: 9262 South 300E, Brookston, IN 47923, US Citizenship: United States	5/25/05
Name: JACKSON, Andrew Indrew Jackson Residence: West Lafayette, IN, United States Mailing Address: 3346 Whirl Away Ct., West Lafayette, IN 47906, US Citizenship: United States	5/25/05
Name: LEMAN, Gregory	
Name: NORMAN, Edward	
Name: KAUFMAN, Robert Residence: St. Louis, MO, United States Mailing Address: 8129 Stanford Ave, St. Louis, MO 63130 Citizenship: United States	

Continuation of Box No. VIII (i) to (v) DECLARATION  If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two inventors are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII" (indicate the item number of the Box) and furnish the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is needed in respect of two or more declarations, a separate continuation box must be used for each such declaration. If this Box is not used, this sheet should not be included in the request.
Continuation of Box No. VIII(iv) Name: CHIEN, John Residence: West Lafayette, IN, United States Mailing Address: 3318 Humbolt Street, West Lafayette, IN 47906, US Citizenship: United States
Name: SHARMA, Vimal
Name: EDWARDS, E. Bradley
Name: HEDRICK, Victoria
Name: JACKSON, Andrew
Name: LEMAN, Gregory 5/24/05 Residence: West Lafayette, IN, United States Malling Address: 3319 Crawford Street, West Lafayette, IN 47906, US Citizenship: United States
Name: NORMAN, Edward
Name: KAUFMAN, Robert

Cuntinualing of Box No. VIII (I) to (v) DECL	AR	TION
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(fthes, sace is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two inventors are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII..." (indicate the leten number of the Box) and furnish the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is needed in rest ect of two or more declarations, a separate continuation hox must be used for each such declaration. If this Box is not used, this sheet should not be included in the request.

Continuation of Box No. VIII(iv)  Name: CHIEN, John
Name: EDWARDS, E. Bradley
Name: HEDRICK, Victoria
Name: JACKSON, Andrew
Name: LEMAN, Gregory
Naine: NORMAN, Edward
Name: KAUFMAN, Robert

# JAP20 Rec'd PCT/PTO 25 JUL 2006

room temperature, filtered, and concentrated under vacuum. The reaction can appear to be decomposing after 6 hours. The bulk solution can be filtered and concentrated under vacuum to a yellow oil (3.3 grams) that can be indentified as

F<sub>3</sub>C F 
$$\frac{CF_3}{F_3C}$$
 F  $\frac{F_3C}{F_3C}$  F  $\frac{EtOH}{AcOH(cat)}$  F  $\frac{F_3C}{F_3C}$  F  $\frac{CHCl3}{30-40^{\circ}C}$  F  $\frac{C}{F_3C}$  F  $\frac{CHCl3}{S3^{\circ}C}$  F  $\frac{CHC$ 

In accordance with scheme (43) above, 5-bromo-1,1,1,2-tetrafluoro-2-trifluoromethylpentane (25 grams) can be dissolved in 25 mL of ethanol and 0.2 mL of acetic acid, and 10.9 grams of potassium thiocyanate can be added to form a mixture. The mixture can be heated to reflux and cooled to room temperature after about 1 to 2.5 hours, and concentrated under vacuum. The concentrate can be partitioned between methylene chloride (100mL) and water (50mL). The aqueous phase can be extracted with methylene chloride (50mL), the organic layers combined, dried over magnesium sulfate, filtered, and concentrated under vacuum to afford a yellow oil that can be identified as 1,1,1,2-tetrafluoro-5-thiocyanato-2-trifluoromethyl-pentane (21.7 grams, 93.9%) by NMR analysis.

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The 1,1,1,2-tetrafluoro-5-thiocyanato-2-trifluoromethyl-pentane can be dissolved in 10 mL of acetic acid and 0.4 mL of water, heated to 40°C and sparged with chlorine. Three additional water (.4mL) treatments can be added every 2 hours with a slight temperature exotherm noted after each addition. The mixture can be sparged and additional water treatments added for a couple of days to result in a heterogenous mixture. The heterogeneous mixture can be partitioned between methylene chloride (100mL) and water (25 mL), the organic layer dried over magnesium sulfate, filtered, and concentrated under vacuum. NMR analysis can indicate 7.1 grams (74.1%) of 4,5,5,5-tetrafluoro-4-

trifluoromethyl-pentanesulfonyl chloride.

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The 4,5,5,5-tetrafluoro-4-trifluoromethyl-pentanesulfonyl chloride (7.1 grams) can be dissolved in 40 mL of chloroform and added to a solution of 8.6 mL of 3-dimethylaminopropylamine in 40 mL of chloroform at 0 °C-5°C drop-wise over 45 minutes (T<sub>max</sub>=5°C) to form a mixture. The mixture can be washed successively with saturated bicarbonate solution (80 mL), water (80 mL), and brine (80mL). The organic layer can be separated, dried over magnesium sulfate, filtered, and concentrated under vacuum to afford 8 grams (93%) of 4,5,5,5-tetrafluoro-4-trifluoromethyl-pentane-1-sulfonic acid (3-dimethylamino-propyl)-amide by NMR and LC/MS analysis.

The 4,5,5,5-tetrafluoro-4-trifluoromethyl-pentane-1-sulfonic acid (3-dimethylamino-propyl)-amide (8 grams) can be dissolved in 25 mL of ethanol containing 3 mL of water and 5.1 mL of 50% (wt/wt) hydrogen peroxide and the resulting solution heated at 35°C for 30 minutes. The reaction can then be allowed to cool to room temperature overnight. Norit, a decolorizing carbon (10 grams) and ethanol (20mL) can be added and the mixture heated to 50°C for 3 hours. The mixture can be filtered through celite, the filter cake washed with 90% (wt/wt) ethanol/ 10% (wt/wt) water (60 mL), and the filtrate concentrated under vacuum, distilled with methanol, and Kugelrohr

to afford 7.1 grams (89.9%) of F<sub>3</sub>C by NMR and LCMS analysis.

$$F_3$$
C  $F_3$   $F_3$ C  $F_3$   $F_3$ C  $F_3$   $F_3$ C  $F_3$   $F_3$ C  $F_3$ 

In accordance with scheme (44) above, 4,5,5,5-Tetrafluoro-4-trifluoromethyl-pentane-1-sulfonic acid (3-dimethylamino-propyl)-amide (6.0 grams) can be dissolved in 25 mL of ethanol containing 1.9 grams of sodium chloroacetate. The resulting solution can be heated to reflux and allowed to reflux for two consecutive nights. After refluxing for approximately 45 hours, the reaction can be stopped, filtered, the salts rinsed and discarded and the filtrate stripped of solvent and identified as

$$F_{3}C$$

$$F_{3}C$$

$$R_{3}C$$

$$R_{4}C$$

$$R$$

In accordance with scheme (45) above, 8-Bromo-1,1,1,2-tetrafluoro-2-trifluoromethyl-octane (20 grams) can be dissolved in 30 mL of ethanol containing 7.6 grams of potassium thiocyanate. Acetic acid (0.2 mL) can be added to form a mixture and the mixture heated to reflux for 4 hours. The mixture can be allowed to cool to room temperature overnight, concentrated under vacuum, and partitioned between methylene chloride (200 mL) and water (100 mL). The organic layer can be dried over magnesium sulfate, filtered, and concentrated under vacuum to afford 18.2 grams (97%) 1,1,1,2-tetrafluoro-8-thiocyanato-2-trifluoromethyl-octane by NMR analysis.

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The 1,1,1,2-tetrafluoro-8-thiocyanato-2-trifluoromethyl-octane (18.2 grams) can be dissolved in 25 mL of acetic acid to form a mixture and the mixture heated to 40°C with chlorine sparging. Initially, 0.8 mL of water can be added to the mixture. Three additional water treatments (0.8mL/each) can be added to the mixture every 2 hours and heated with the chlorine sparge continued overnight and an additional 0.8mL of water added, the mixture can be cooled and partitioned between methylene chloride (200mL) and water (100mL). The aqueous layer can be extracted with methylene chloride (100mL). The organic layers can be combined, washed three times with water (100mL/each), dried over magnesium sulfate, filtered, and concentrated to yield 19.5 grams (94.5%) of 7,8,8,8-tetrafluoro-7-trifluoromethyl-octanesulfonyl chloride by NMR analysis.

The 7,8,8,8-tetrafluoro-7-trifluoromethyl-octanesulfonyl chloride (19.5 grams) can be dissolved in 100 mL of chloroform and added to 20.9 mL of dimethylaminopropylamine in 100 mL of chloroform at 0 °C-5°C over 1 hour to form a mixture. When the addition is complete, the mixture can be allowed to warm to room temperature and can be stirred at ambient for one hour. The mixture can be washed twice with saturated bicarbonate solution (100mL/each), deionized water (200mL), and brine (200mL). The organic layer

solution (100mL/each), deionized water (200mL), and brine (200mL). The organic layer can be dried over magnesium sulfate, filtered, and concentrated under vacuum to afford a yellow oil that can be identified as 7,8,8,8-tetrafluoro-7-trifluoromethyl-octane-1-sulfonic acid (3-dimethylamino -propyl)-amide (24.09 grams, 95.97%) by NMR.

The 7,8,8,8-tetrafluoro-7-trifluoromethyl-octane-1-sulfonic acid (3-dimethylamino-propyl)-amide (7 grams) can be dissolved in 25 mL of ethanol containing 2.3 mL of water and 4.0 mL of 50% (wt/wt) hydrogen peroxide and the resulting solution can be heated at 35°C overnight. Decolorizing carbon (8 grams) and ethanol (15mL) can be added to the solution and the solution heated to 50°C for three hours. The solution can then be cooled to room temperature, filtered through celite, the filter cake washed with 90% (wt/wt) ethanol/deionized water (50mL), and the filtrate concentrated under vacuum to a wax like solid. The solid can be distilled twice with ethanol to afford a yellow oil that can be placed on a Kugelrohr for two hours at 40°C and 0.1 Torr to afford a white solid (5.9 grams, 79.9%) of

$$F_3C$$
 $F_3C$ 
 $F_3C$ 
by NMR analysis.

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F<sub>3</sub>C ONa EtOH

Reflux

$$F_3C$$
 $F_3C$ 
 $F_3C$ 

In accordance with scheme (46) above, 7,8,8,8-tetrafluoro-7-trifluoromethyloctane-1-sulfonic acid (3-dimethylamino-propyl)-amide (6.0 grams) can be dissolved in 25 mL of ethanol containing 1.6 grams of sodium chloroacetate. The resulting solution can be heated to reflux and allowed to reflux and stir over for 40 hours. The solution can be quenched, filtered, the solvent stripped, and the resulting solid placed in a drying oven (50°C, 1 Torr) overnight. The remaining solids can be identified as

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In accordance with scheme (47) above, 2-(3-Bromo-propoxy)-1,1,1,3,3,3-hexafluoro-propane (19 grams) and potassium thiocyanate (8.3 grams) can be dissolved in 30 mL of ethanol containing 0.2 mL of acetic acid and heated to reflux. After 2.5 hours at reflux, the reaction mixture can be cooled to room temperature and concentrated under vacuum to a semi solid. The semi solid can be partitioned between ether (100mL) and deionized water (100mL). The organic layer can be dried over sodium sulfate, filtered, and concentrated under vacuum to afford a yellow oil (16.88 grams, 90.3%). The yellow oil can be identified as 1,1,1,3,3,3-hexafluoro-2-(3-thiocyanato-propoxy)-propane by NMR.

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The 1,1,1,3,3,3-hexafluoro-2-(3-thiocyanato-propoxy)-propane (16.9 grams) can be dissolved in 30 mL of acetic acid and 0.8 mL of water to form a mixture. The mixture can be heated to 40°C and sparged with chlorine. The mixture can then be treated three times with deionized water (0.8mL) every two hours, and the mixture heated to 40°C under a chlorine sparge for about 48 hours. The mixture can be allowed to cool to room temperature, partitioned between methylene chloride (100mL) and deionized water (100mL), the organic layer separated and washed three times with deionized water (100mL/each), dried over magnesium sulfate, filtered, and concentrated under vacuum to a colorless oil 3-(2,2,2-trifluoro-1-trifluoromethyl-ethoxy)-propane-1-sulfonyl chloride (18.4 grams, 99.3%) by NMR.

The 3-(2,2,2-trifluoro-1-trifluoromethyl-ethoxy)-propane-1-sulfonyl chloride (18.4 grams) can be dissolved in 100 mL of chloroform and added to 22.5 mL of dimethylaminopropylamine in 100 mL of chloroform 0 °C-5°C over 1 hour to form a mixture. When the addition is complete the mixture can be allowed to warm to room temperature and stir at ambient for 1 hour. The mixture can be washed with saturated bicarbonate solution (200 mL), deionzied water (200mL), and brine (200mL). The organic layer can be dried over magnesium sulfate, filtered, and concentrated under vacuum to afford a yellow oil that can be placed on the Kugelrohr 15 minutes at ambient temperature and 0.1 Torr to afford 3-(2,2,2-trifluoro-1trifluoromethyl-ethoxy)-propane-1-sulfonic acid (3-dimethylamino-propyl)-amide (20.88g (92.8%)) by NMR.

The 3-(2,2,2-trifluoro-1-trifluoromethyl-ethoxy)-propane-1-sulfonic (3acid dimethylamino-propyl)-amide (7 grams) can be dissolved in 25 mL of ethanol containing 2.6 mL of water and 4.4 mL of 50% (wt/wt) hydrogen peroxide to form a mixture and the mixture heated at 35°C overnight. Decolorizing carbon (8 grams) and ethanol (15mL) can be added to the mixture, the mixture heated to 50°C for 3 hours, filtered through celite, the filter cake washed with 90% (wt/wt) ethanol/water (50mL) and the filtrate can be concentrated under vacuum to afford a white semi-solid. The solid can be refluxed twice in ethanol prior to being Kugelrohr 40°C placed on the for hour at and 0.1 Torr afford to

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$$F_3C$$
 $CF_3$ 
 $(6.66 \text{ grams (90.0\%)}) \text{ by NMR.}$ 
 $F_3C$ 
 $CI$ 
 $ONa$ 
 $EIOH$ 
 $Reflux$ 
 $F_3C$ 
 $CF_3$ 
 $ONa$ 
 $ONa$ 

In accordance with scheme (48) above, 3-(2,2,2-trifluoro-1-trifluoromethyl-ethoxy)-propane-1-sulfonic acid (3-dimethylamino-propyl)-amide (6.0 grams) can be dissolved in 25 mL of ethanol containing 1.9 grams of sodium chloroacetate. The resulting solution can be refluxed and stirred for 40 hours, the reaction quenched, and filtered. The solvent can be stripped and the resulting solid placed in a drying oven (50 °C, 1 Torr) overnight to

$$F_3C \longrightarrow O \longrightarrow N \longrightarrow O \oplus O$$
 yield  $CF_3$  by NMR.

For example and by way of example only,  $\ddot{b}$  be a  $Q_{FS}$  portion.  $R_F$  stabilizers can be prepared according to scheme (59) below.

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Referring to scheme (59) above, potassium carbonate (2.37 grams), methioglycolate (1.82 grams) and dimethylformamide (DMF) (20mL) can be added and the mixture heated to  $50^{\circ}$ C for 3 hours. The mixture can be allowed to stir overnight at room temperature to form a yellow slurry which can be added to water (50mL) and ethyl acetate (50mL), the organic layers combined, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and stripped of solvent.

In a nitrogen atmosphere, thioester (4.0 grams) and polyethylenimine (PEI, mw=1200) (5.3 grams) can be placed in isopropanol (5mL) and stirred until dissolved to form a mixture. Sodium methoxide (0.15 grams) and sodium borohydride (0.04 grams) can be added to the mixture and the mixture heated to 115°C for 15 hours, then stirred at room temperature for 2 days. Removal of remaining isopropanal can be difficult. A solution of sodium chloroacetate (10.52 grams) in water (25mL) can be added drop-wise to the mixture and the temperature kept below 55°C and the mixture then heated to 70°C for two hours. NaOH (1.23 grams of a 50% (wt/wt) solution of NaOH and water) can be added to raise the pH of the mixture to at least 7.5 from the starting pH of approximately 6. The mixture can then be allowed to continue stirring at 70°C for 2 additional hours, the heat then removed,

and the resulting 
$$F_3C$$
  $F_3C$   $F_3$ 

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# 10/587444 AP20 Rec'd PCT/PTO 25 JUL 2006

### PATENT COOPERATION-TREATY

INTERNATIONAL APPLICATION SERIAL NO	PCT/US05/03138
FILING DATE	.28 January 2005 (28.01.2005)
APPLICANTS	
AGENT'S FILE REFERENCE	PC3-160
TITLE: Production Processes and Systems, Compositions,	
Metal Complexes, Phosphate Esters, Glycols, Aqueous Fil	
Stabilizers	3

# LETTER REGARDING AMENDED REQUEST

To:

Claudine PERGOD

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## **REMARKS**

Applicant submits herewith replacement sheets 1, 2, 3, 5, 6, 7 and 8 of the originally filed Request. Sheet 1 has been amended to correct Inventor Boggs' address, to correct Applicant designations, and to amend Agent's fax number. Sheet 2 has been amended to correct Applicant designations. Sheet 3 has been amended to correct Applicant designations and to correct Inventors Jackson and Norman's addresses. Sheet 5 has been amended to include date of assignments, correct Inventors Jackson and Norman's addresses and to amend wording to meet the prescribed wording. Sheet 6 has been amended to incorporate date of assignments and to amend wording to the prescribed wording. Sheet 7 has been amended to include Inventor Boggs' signature and date. Sheet 8 has been amended as sheets 8a-d to include inventors' signatures and dates.

Please replace sheets 1, 2, 3, 5, 6, 7 and 8 of the original Request with the enclosed replacement sheets.

Applicant further submits replacement sheets 68-73, 91 and 106 of the originally filed specification. Sheets 68-73 and 106 have been amended for clerical purposes. Sheet 91 has been amended to correct an error in a depicted compound.

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# JAP20 Rec'd PCT/PTO 25 JUL 2006

Please replace sheets 68-73, 91 and 106 of the originally filed specification with the enclosed replacement sheets.

Respectfully Submitted,

Date: 5/27/05

By:

HYTA, Robert C.

Reg. No.: 46,791